2410-2E2 (2nd Quarterly Report)

~~Second~~ Quarterly Report

INVESTIGATION OF THE REINFORCEMENT OF DUCTILE METALS WITH STRONG, HIGH MODULUS DISCONTINUOUS, BRITTLE FIBERS

by

A. Gatti, J.M. Berry, J.J. Gebhardt, E. Feingold

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINSITRATION

May 1967

CONTRACT NASw-1543

Space Sciences Laboratory
GENERAL ELECTRIC COMPANY
Missile and Space Division

CONTENTS

ILLUSTRATIONS

SUMMARY

This program report covers the period 1 February 1967 to 30 April 1967; the work is being performed under Contract NASw-1543, with Mr. James J. Gangler of NASA-Headquarters serving as Program Monitor.

The purpose of this program is to define and investigate the critical factors affecting the reinforcement of ductile metals with short, brittle fibers. The materials systems selected for study are aluminum (or its alloys) and "ductile" epoxies reinforced with B_4C whiskers or with high modulus filaments, such as, $B_4C/B/W$, SiC/W, B/W, etc. Related tasks in the program include the development of a more economical process for growing B_4C whiskers, deposition studies, the production of B_4C fibers, and the characterization of the individual constituents in the final composites. The latter task involves a study of the structural and chemical interactions of the combined elements (fibers, matrix, coatings, etc.).

The results obtained during this period are summarized as follows:

(1) $B_4C/B/W$ filaments have been made by depositing B_4C from gaseous mixtures of methane, hydrogen and boron trichloride under conditions similar to those used previously in another program.[1] A new technique of using 0.004"-diameter B/W filaments as the substrate for depositing a B_4C outer layer has resulted in $B_4C/B/W$ in diameters greater than 0.005". These filaments will be used in future studies rather than the 0.0025" diameter filaments of $B_4C/B/W$ used previously. Thus far, strength values of 234,000 psi* have been measured for the new larger-diameter filaments.

(2) A fabrication procedure has been developed which results in a ductile matrix material from hot-pressed aluminum powder (1100 alloy). This process provides another means for fabricating aluminum-fiber composites as an alternative method for evaluating aluminum single-filament composites.

(3) Individual filaments of $B_4C/B/W$ having 0.1" gauge lengths were pulled in tension. A high value of 540,000 psi was measured for the best filaments. A statistical study of strength vs. gauge length was also extended to include filaments of SiC/W. It was found that this material has a much narrower

* The data are based on 1" gauge lengths.

scatter of tensile strength values among samples and as a function of gauge length. The highest tensile strength value obtained for the SiC/W filament was 425,000 psi for a 0.1" gauge length.

(4) An epoxy formulation based on Novolac (DEN 438) was developed which has the desirable characteristics of both ductility and good bond strength for filaments of B_4C/B/W, SiC/W and B/W. The formulation and sample preparation techniques are described.

(5) An extensive study of the fracture behavior of epoxy-filament composites in tension has been made. It is shown that the rule of mixtures theory is inadequate to describe the failure behavior of these composites, since a change in fracture mode is observed at a critical strain rate. These effects are discussed for specimens reinforced with either discontinuous or continuous filaments.

(6) The room temperature tensile tests of single filament B_4C/B/W-aluminum composites, discussed in the First Quarter[2] of this program were repeated for both B_4C/B/W and SiC/W filaments using a powder metallurgy approach. It was concluded that the powder process now being used compares favorably with a formerly used process.

(7) Aluminum-SiC/W continuous-filament composites were fabricated by liquid infiltration at 5, 10, 20, 40, and 60 volume percent filaments. It was found necessary to coat the filaments with Ti/Ni sputtered coatings to insure complete infiltration. Unfortunately, titanium appears to enhance the diffusion of silicon to the tungsten core of the SiC filament. As a result, the filaments were weakened considerable. Strengthening efficiencies averaged only about 25% of the potential loading of these fibers.

(8) A study of the fracture mode of all aluminum-SiC/W composites showed that all filaments broke in essentially the same plane of fracture with no random array of filament failure evident anywhere else in the composites. A random behavior would be expected if no dynamic effects occured. However, these studies indicate that once a filament breaks, the composite fails, which suggests that an autocatalytic effect is operating. These results are described in greater detail in this report.

I. INTRODUCTION

From a reinforcing viewpoint, whiskers (single-crystal filaments) appear to have many desirable characteristics. A number of classes of compounds have been prepared in this form including metals, oxides, nitrides, carbides, and graphite. The maximum strengths observed for these whiskers range from about 0.05 to 0.1 of their elastic moduli, approaching predicted theoretical strengths. Many also have relatively low densities and are stable at high temperatures. Calculations of whisker-reinforced composite properties based on whisker properties, particularly for the brittle whiskers of high modulus materials, show that they have enormous potential compared to more conventional materials on both a strength/density and a modulus/density basis.

The incorporation of whiskers into composites requires the following series of processing steps:

1) Whisker growth
2) Whisker beneficiation, to separate strong fibers from the growth debris
3) Whisker classification, to separate according to size
4) Whisker orientation, to align the whiskers and maximize reinforcement along a specific axis
5) Whisker coating, to promote wetting and bonding
6) Whisker impregnation with matrix material, to form a sound strong composite.

Because of the many processing steps, there is a large number of imposing technical problems to be solved in order to achieve the high potential strengths. Many of these problems have not been solved yet.

In a few isolated cases, involving very small and carefully prepared samples, the predicted strengths of the brittle whisker/ductile matrix composites have been achieved. However, all too frequently, attempts to scale up the composites into even modest size specimens have resulted in strengths that range from about 10 to 30 per cent of the predicted values.

A list of possible reasons for the low composite strength values is given in Table I. As can be seen, there are many variables to contend with, and many of these are interrelated and difficult to study experimentally.

TABLE I. VARIABLES AFFECTING THE TENSILE STRENGTH OF WHISKER REINFORCED COMPOSITES

A. Whisker Variables

1. Average strength
2. Dispersion of strength values
3. Strength versus whisker diameter and length
4. Strength degradation during handling and fabrication
5. Strength versus temperature
6. Modulus

B. Matrix Variables

7. Yield strength
8. Flow properties
9. Strength versus temperature (particularly shear strength)
10. Matrix embrittlement due to mechanical constraints on new phases formed.

C. Composite Variables

11. Volume fractions of components--fiber & matrix
12. Homogeneity of whisker distribution

A fundamental difficulty in evaluating the performance of whisker composites is the lack of knowledge concerning the whiskers themselves. This is understandable when one realizes that there are about 10^9 to 10^{10} of them per pound, and characterization of even a small fraction becomes a major task. These and other problems have limited the immediate use of B_4C whiskers which were synthesized and characterized in previous studies (3, 4, 5, 6).

An alternate means to gain useful, fundamental knowledge concerning whisker-reinforced composites involves the use of brittle, continuous filaments.

Continuous filaments have several advantages over whiskers when investigating the reinforcement of materials; some of these advantages are listed below:

1) It is much easier to characterize the relevant and critical parameters listed in Table I.

2) The available continuous filaments are large relative to the whiskers and can be more readily handled and incorporated into composites.

3) The filaments can be cut to uniform, desired lengths of symmetrical geometry so that the effects of discontinuous reinforcements can be assessed.

Experimental work of this type has already been done using ductile filaments such as tungsten in a ductile matrix such as copper[7]. Although this work has provided a wealth of information regarding the reinforcement of metals, it does not uncover all of the key problems encountered in the brittle fiber/ductile matrix systems which are potentially of great technological importance. The chief difference between the reinforcement of metals with brittle and ductile fibers, is that ductile fibers can deform to accommodate local, high stress concentrations, whereas brittle fibers cannot do so. Thus, it is necessary to carry out further studies and to evaluate the potential and engineering limitations of metals reinforced with brittle fibers and whiskers.

This program was therefore initiated to investigate in detail the behavior of a ductile metal (aluminum) reinforced with various brittle fibers, such as, $B_4C/B/W$, SiC/W, B/W, etc. (both continuous and chopped), to provide data which would be pertinent to whisker-reinforced metals. This program is being conducted in two parts: (1) development of a process to grow B_4C whiskers which would be amenable to eventual scale-up, and (2) to investigate the reinforcement of aluminum with brittle, high modulus filaments. When sufficient quantities of whiskers become available, they will be used in the composite studies also.

II. EXPERIMENTAL PROCEDURES - RESULTS AND DISCUSSION

A. B_4C FILAMENT DEPOSITION AND WHISKER GROWTH STUDIES

During the present report period, attention has been directed primarily toward the preparation of boron carbide filaments having sufficiently large diameters to permit exploration of filament diameter variables in single-filament composites and also to allow easier achievement of high filament volume fractions in multiple filament composites. Conditions have been established for deposition of boron carbide on preformed 4-mil tungsten-core boron filaments as well as on tungsten alone (0.5 mil). Modifications in the method of heating are required however, for continuous, reliable production. These are in progress and production is expected to commence shortly.

1. Boron Carbide Continuous Filaments

Up to the present time, the boron carbide filaments used in composite preparation have consisted actually of a layer of boron carbide, saturated with vitreous carbon, deposited over a boron undercoating on a tungsten substrate. The initial reason for the boron coating was to prevent the occurrence of 'hot spots' on the filament which caused the filament to break during deposition. Another reason for the boron undercoating, however, was to increase the diameter of the finished filament by acting as a high strength, relatively high modulus filler as it were, beneath the boron carbide layer. The need for such a filler exists because of the relatively low deposition rate of boron carbide[8] compared to the higher rate for deposition of boron alone. The former is apparently diffusion controlled, while the latter, in the proper reactor geometry, can be carried out at the maximum rate allowed by the chemical reaction kinetics. Therefore, while boron filaments of significant thickness (e.g., 3-4 mils) can be built up in a reasonable period of time, boron carbide filaments can be made only at low production rates.

In order to prepare boron carbide filaments of desirable diameter (greater than 4 mils), commercially available tungsten-core boron filament was used as the initial substrate for deposition of boron carbide from boron trichloride, methane and hydrogen gas mixtures.

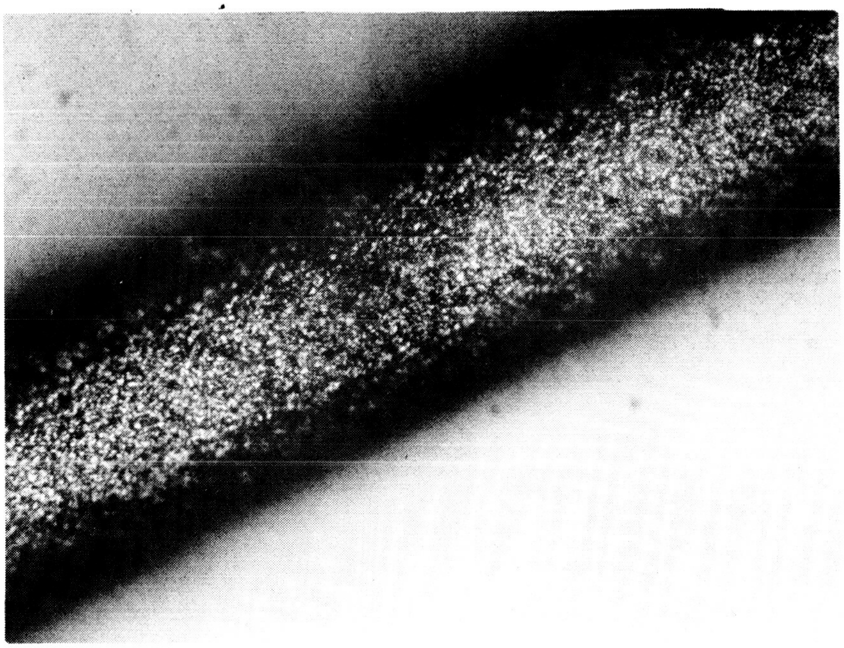
The primary difficulty in carrying out the deposition in this manner lay in heating the boron filament to deposition temperature and maintaining it at the desired level during deposition. The boron filament substrate consists of a semiconducting boron layer deposited on a conducting borided tungsten core. Cold resistance of the filament is about 30,000 ohms/inch, and the path of conduction is believed to be through the core. When the tungsten core boron filament is at deposition temperature in an inert atmosphere, steady and uniform temperature profiles are easily achieved using about twelve hundred volts at a few milliamperes from an A.C. transformer. However, as soon as boron carbide begins to deposit, the surface apparently becomes conductive, resistance drops and the filament requires only a few hundred volts, while the current rises to over two hundred milliamperes. Adjustment is quite critical around this crossover point to prevent overheating and breaking the filament, and little uniform filament can be made with this type of heating. At present, a power supply suited to this application is being constructed and will be available in the near future. However, some filament was prepared, the properties of which are described below and indicate that it will be useful for the purpose intended on this program.

It was also found that in the apparatus now being employed, boron carbide can be deposited directly on a tungsten core without the difficulty due to hot spots encountered in earlier work.[7] The deposition rate is still low however, and filaments produced in this manner will be considerably thinner than those having a boron undercoating. Properties of this material are also described below.

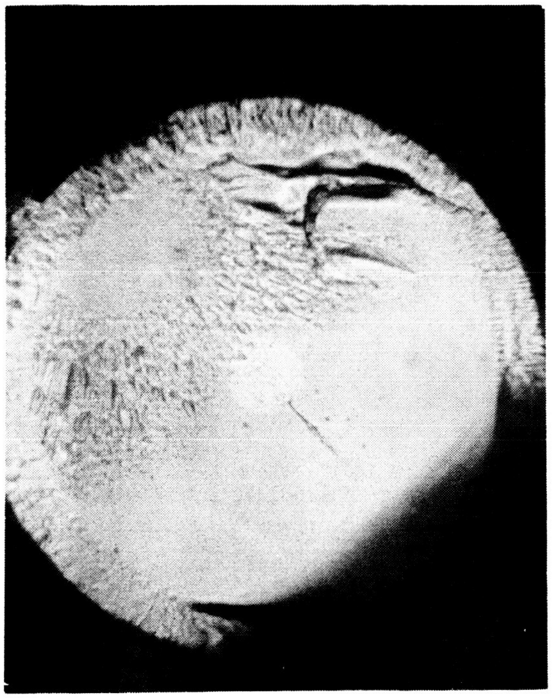
2. Filament Properties

(a) Boron Carbide on 4-mil Boron/Tungsten

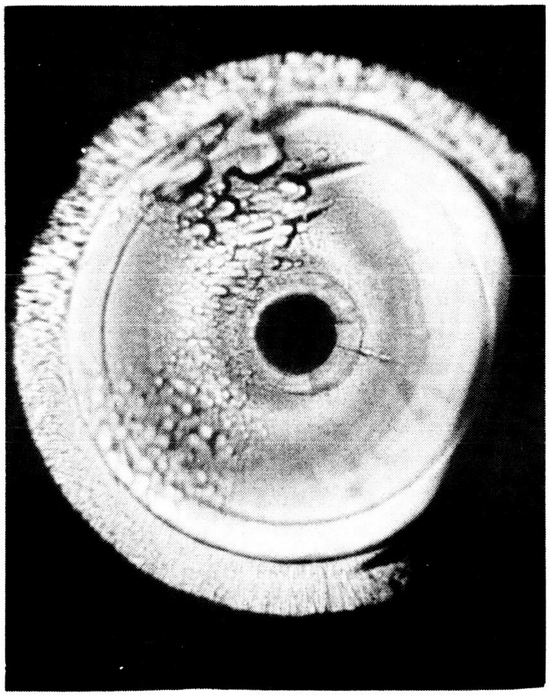
Despite the difficulties encountered in maintaining steady temperatures during deposition, sufficient material was produced to permit preliminary characterization. Figure 1 shows the surface and cross section of this product. The outer layer is approximately 0.5 mil thick making the total diameter 5 mils, and the volume of boron carbide around 37%. Etching



A. SURFACE VIEW (420 X)



B. FRACTURE SURFACE (593X)



C. FRACTURE SURFACE AFTER ETCHING (HOT 50% H_2O_2) (593 X)

Figure 1. Boron Carbide (5 mil total diameter) Deposited on 4 mil B/W Filament

results and electrical conductivities both indicate that the outer layer is essentially the same as that produced in the earlier work in which this filament was developed.[9] X-ray diffraction patterns also confirm the existence of boron carbide, but of somewhat higher crystallinity than the earlier material. The surface also appears to be identical to that characteristic of boron carbide deposited on a freshly formed boron substrate. Mechanical properties (tensile strength and elastic modulus) were determined on somewhat un-representative specimens as well as on the substrate as received and on the substrate heated to deposition temperature in an inert atmosphere. These results are shown in Table II.

Table II

Mechanical Properties of Boron Carbide/4-mil Boron/Tungsten

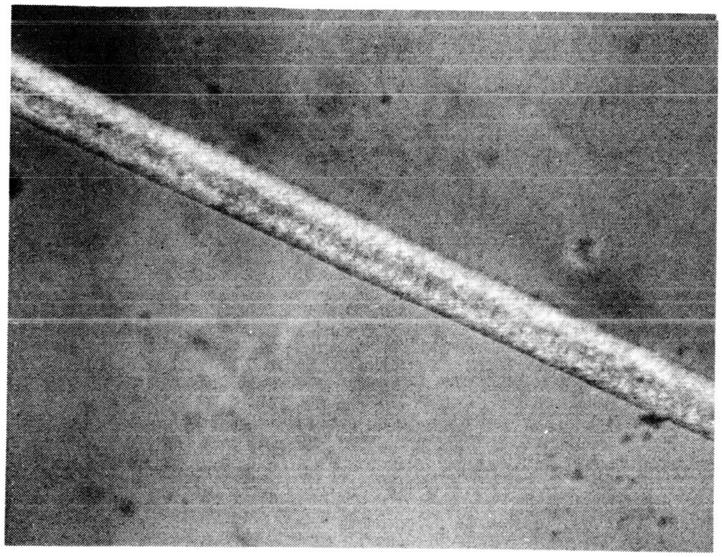
Material	Tensile Strength*	Modulus
Boron/tungsten, as rec'd (4 mil)	241,000 psi	53
Boron/tungsten, heated (4 mil)	104,000	--
Boron carbide/boron/tungsten (5 mil)	234,000	--

*Avg. of Five Tests

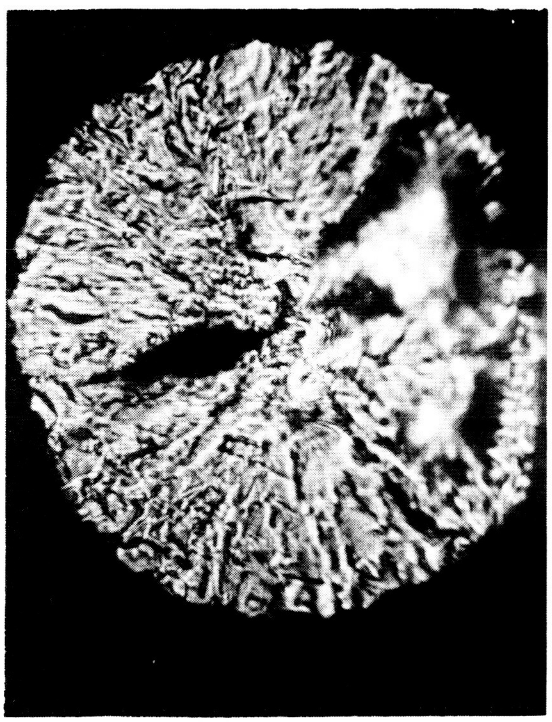
Little more can be said at this early time about the mechanical properties of the filaments except that the weakening of the substrate during heating is not unexpected, since the original deposition temperature for boron was near 1100°C, and structural rearrangements have been observed in such cases before.[9]

(b) Boron Carbide on Tungsten

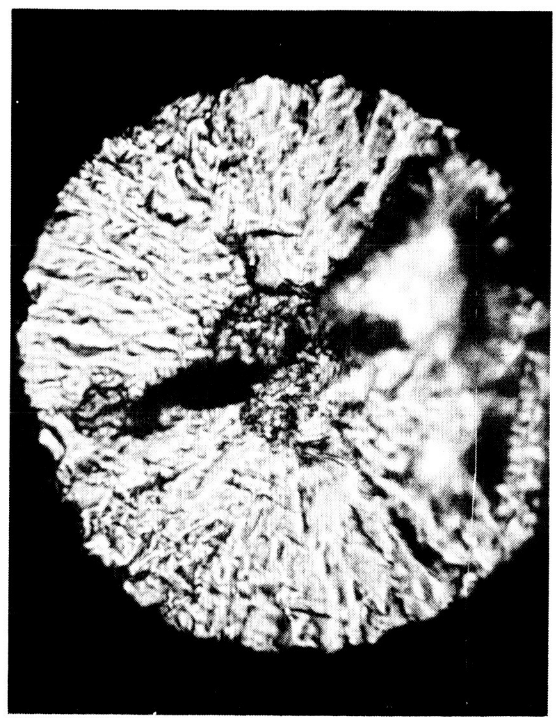
Some filament was also prepared using only a tungsten substrate. Figure 2 shows the cross sectional and surface appearance. The large void which appears to start at the core, and the two incipient cracks at about right angles to this void are also characteristic of boron/tungsten filaments and are presumably due to the existence of residual tensile stresses at the core/sheath interface. Strengths were quite low for this material (ca. 5,000 psi) which is not unusual considering the defects. The modulus was also low (ca. 25 million psi) and may indicate that an excess of carbon



A. SURFACE VIEW (116X)



B. FRACTURE SURFACE (1210X)



C. FRACTURE SURFACE AFTER ETCHING (HOT 50% H_2O_2) (1210X)

Figure 2. Boron Carbide on Tungsten Substrate, Diameter 2.25 mil.

was deposited over that percentage at which maximum strength and modulus are observed.[1]

3. Boron Carbide Whiskers

Additional attempts were made to produce boron carbide single crystal whiskers by the simultaneous reduction of boron tribromide and carbon tetrachloride at temperatures near 1400°C. Several improvements have been made in the deposition system primarily to eliminate possible sources of failure. These include a more efficient trapping system to prevent the back diffusion of hydrogen bromide which could reverse the reduction equilibria, and the addition of vanadium tetrachloride (anhydrous) to the mixture of halides used as the feed material. Vanadium had been found to be a nucleation initiator in the evaporation method for growth of whiskers.[3] Despite these innovations however, the results have continued to be discouraging. In most of the experiments, boron carbide thin films have formed, together with some dendritic overgrowths. The scarcity of the latter is indicative of a nucleation problem rather than a difficulty in producing deposition species.

In the absence of adequate fundamental information concerning nucleation, an empirical approach appears to be the most practical; this would consist of carrying out alternating evaporation and vapor deposition experiments until the critical nucleation factors can be isolated. It will be recalled from an earlier report[2], that the use of furnace components previously involved in evaporation experiments was believed to have been a possible reason for success in growing whiskers by vapor deposition in the first attempt, followed by repeated failure after that time.

B. CHARACTERIZATION OF COMPOSITE MATERIALS

Since this program is concerned with an investigation of the factors which control the mechanical, the physical and the chemical behavior of metal-matrix composites reinforced with brittle, discontinuous fibers, it is highly important that parameters which affect this behavior be well identified and characterized. The approach used evolves from the simple concept of combining well-characterized brittle fibers with a well-characterized matrix metal using simple composite test configurations.

The variables include such factors as the average strength and strength dispersion of the fibers, fiber aspect ratio (L/d), fiber strength degradation during processing, and so forth. After systematically varying composite parameters and comparing the results with existing theory, the theory will be either verified or modified to account for the experimental observations. In this manner the key variables and their relative importance should be clearly defined.

1. Matrix Characterization

Commercially pure aluminum (1100 alloy) is being utilized as the metal matrix material because it has the following advantages:

1) Its properties are well documented
2) It is a ductile metal
3) It can be purchased in almost any form including sheet, tubing, powder, etc.
4) Composite specimens can be fabricated by several techniques.

The method used previously[2] for fabricating single filament $B_4C/B/W$-Aluminum Composites utilized the technique of hot pressing an assembly of sheets and tubes and filaments to form a specimen approximately 1/8" square by 4" long. After tensile testing the composites, the aluminum matrix was dissolved away, the number of broken pieces counted, and their fracture surfaces were examined. A question as to the validity for using single-filament test specimens arose, because an imbedded filament could conceivably slide within the tube during straining because of poor bonding, thereby giving a false indication of its estimated strength when the relationship $L/d = \sigma/2\tau$ is used. Therefore a powder processing technique was substituted which produced a fully dense, ductile specimen of identical size to permit checking this possible discrepancy. Processing variables found necessary to produce such a specimen were to use aluminum powder MD grade 101 (-100 mesh)* pressed at 550°C and 10,000 psi for 20 minutes. This modified specimen preparation method was used for all subsequent single filament-aluminum composite studies.

*Metals Disintegrating Co., Elizabeth, N. J.

2. Filament Evaluation

The strength properties of composites containing high modulus, high strength, brittle fibers are primarily dependent on the fiber properties. Therefore, it is essential to measure the strength characteristics of the fibers both before and after fabrication into composites.

A previous report[2] contained a brief summary of the benefits to this program of using $B_4C/B/W$ filaments as a substitute system for B_4C whiskers until such time as whisker technology could be relied upon to produce acceptable material in large quantities at reasonable cost. It is to be realized that other high performance filaments such as B/W and SiC/W can also be of benefit to this program. Accordingly, SiC/W filament* was purchased so that its reinforcing behavior could also be evaluated along with the $B_4C/B/W$ filaments made in this Laboratory.

Previous work[2] had raised the possibility that very short fibers of $B_4C/B/W$ could have strengths in the 600,000 psi range. This was studied further by extending the tensile testing of $B_4C/B/W$ filaments to gauge lengths of 0.1". Figure 3 is a summary of these results and shows an indication that for $B_4C/B/W$ material at least, a variation in strength occurs as a function of gauge length. Also included in Figure 3 is a summary of the tensile strength of SiC/W filament as a function of gauge length. It can be seen that the strength of this material is not as dependent upon gauge length as the $B_4C/B/W$ material. Figure 4 shows a typical fractograph of SiC/W, and indicates that the origin of fracture of this material always occurs from the core area. It is hypothesized that this behavior of SiC/W arises in a similar fashion to that attributed to cast iron, which is influenced by an infinite number of internal notches so that its fracture stress lies in a narrow band of values. Filaments of $B_4C/B/W$ however, seems to exhibit a more variable behavior because of a more complex state of high internal stresses.[11]

It is also highly desirable to evaluate the strength of filaments after they have been processed into composites to ascertain if they have suffered any damage which would weaken the composite specimens. Three 2" long

*General Technologies Corp., Reston, Va.

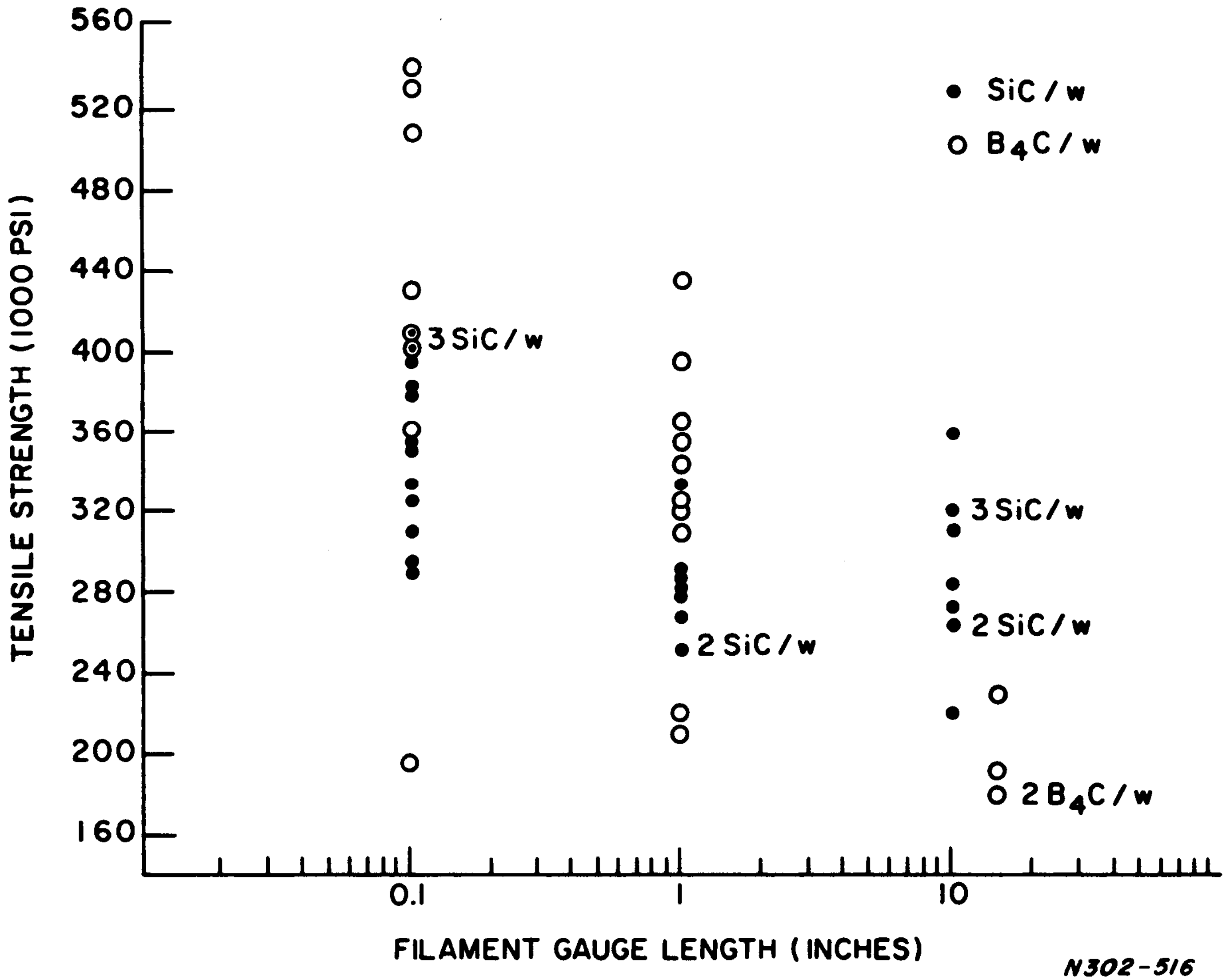
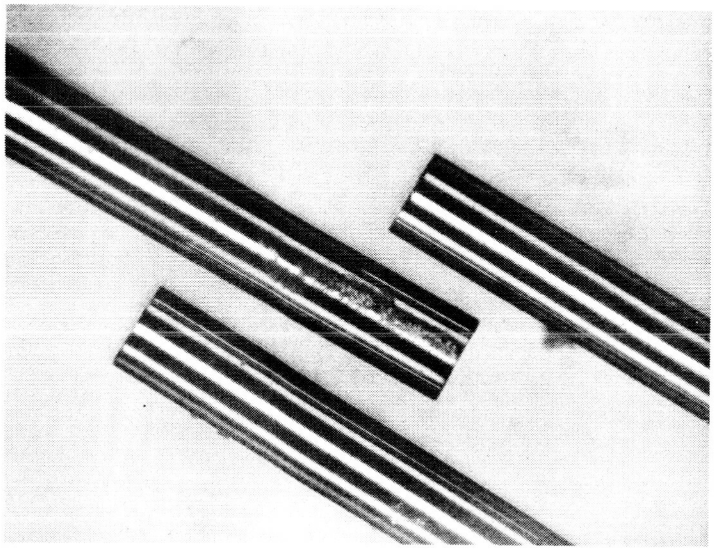
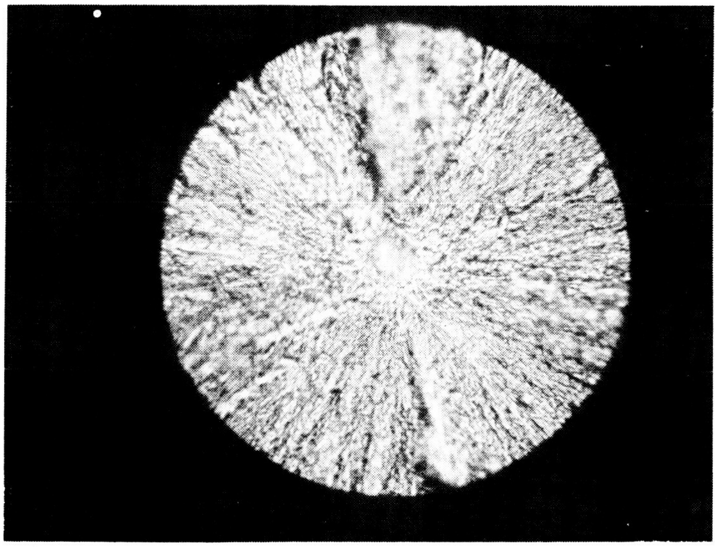


Figure 3. Room Temperature Tensile Strength vs. Gauge Length for $B_4C/B/W$ and SiC/W Filaments.



A. LONGITUDINAL VIEW – NOTE SQUEEZE ENDS AND SMOOTH SURFACE 116X



B. FRACTURE CROSS-SECTION – RADIAL MARKINGS INDICATE FRACTURE ORIGIN IN CORE OR AT CORE SHEATH INTERFACE. 593X (d = 0.0041)

Figure 4. Typical Fracture Mode of SiC/W Filaments.

filaments of $B_4C/B/W$ were recovered intact from single filament-aluminum composites by etching in 50-50 HCl -H_2O. Tensile tests of these filaments indicated that they had been weakened, since the filaments only averaged 60,000 psi. However, it was previously shown that in aluminum composites, the original strength of the filaments is retained.[2] Thus no positive explanation of these results can be made at the present time. A tentative conclusion would be that the etchant was harmful or that an insufficient number of filaments was tested because of the scatter shown in Figure 3. These results will be studied further in future work.

C. COMPOSITE STUDIES

1. Epoxy-Fiber Studies

(a) Composition Studies and Fabrication Variables

The incorporation of brittle filaments into an epoxy matrix which approximates aluminum metal in its mechanical behavior would offer certain advantages because of its transparency. A search was made of epoxy systems and a formulation attributable to V. Mazzio[12] was finally standardized. The epoxy formulation is shown in Table III. Formulations which were softer than the "standard" material (by the addition of more plasticizer) had poor bonding strength, while harder material was barely "ductile" and consistantly failed at the first filament break. (This phenomena is discussed further in the following sections of this report). The curing cycle and mold release agent were also important variables which had to be controlled in order to consistently duplicate the epoxy-filament tensile behavior. Epoxy-filament composites were fabricated using the following processing stages:

(1) A shallow steel dish was sprayed with MS-122 Fluorocarbon Dry* mold release and a thin (1/16" thick) layer of standard epoxy was laid down and cured at 180°F for 2 hours. It is important to mention that other release agents such as RAM 225** (solvent solution of resins) were tried but, because these resinous film type materials are somewhat mobile, the material would diffuse to the top of the first layer of epoxy and disrupt the subsequent bonding

* Miller-Stephenson Chemical Co., Phila., Pa.
** RAM Chemicals, Gardena, California

Table III

Formulation for Epoxy Novolac Composition

Material	Chemical Name	Symbol Used	Quantity Parts
Basic Epoxy	Epoxy Novolac	DEN 438	52
Plasticizer	Polypropylene Glycol	PPG 425	30
Curing Agent	Methyl Nadic Anhydride	MNA	36
Cure Promoter	Benzyldinethylamine	BDMA	1

of the second layer. Also the Fluorocarbon dry mold release allowed a slight bonding of the epoxy to the steel dish so that curing the shrinkage was confined to the thickness of the cured epoxy layers thereby eliminating compressive straining at the epoxy filament interface in the longitudinal direction. RAM 225** however, allowed the epoxies to shrink in the longitudinal direction which always led to premature partial debonding of the filaments.

(2) After the first cure, a second layer of epoxy of the same thickness as the first was laid down and filaments placed into the liquid layer in any geometric pattern desired.

(3) The sample was cured an additional hour at 180°F and then post cured 2 hours at 350°F without removal from the oven.

(4) The epoxy cake was released from the mold by freezing and tapping. Specimens were sawed from the cake and ground to the shape of dog bone tensile bars for subsequent tensile testing.

(b) Tensile Test Results of Single Filament Epoxy Composites

The results to be described and discussed in this section show that the fracture mode, the strength, and the "ductility" of single filament-epoxy composites are markedly affected by the strain-rate and prior strain history of the specimens. Previous attempts to get at such effects had been made using SiC/W filaments but were frustrated by inability to get the strain rate sensitivity of the epoxy to straddle the available (machine limited)

**RAM Chemicals, Gardena, California

strain rates. Fortunately, results from another program (concerned with determining filament-surface differences relevant to bonding) provided excellent examples of the effect of some of the variables under study in this program.

Four tensile tests were made on epoxy specimens of this type, each containing a single boron-tungsten filament. Fig. 5 is a schematic representation of the specimen configuration which may be described as an unreinforced discontinuous composite. Two of the boron filaments were manufactured by AVCO and two by T.E.I.*, and the different sources seemed irrelevant to the results. Two specimens were tested at a strain rate of approximately 0.8 in/in/minute. The other two specimens were pulled at 0.008 in/in/minute until maximum loading was reached. After visual examination to determine the number of cracks in the filament, these same specimens were again pulled in tension at the higher strain rate (0.8). Table IV contains pertinent test data and Figure 6 shows the stress-strain curves at the higher strain rate for virgin and prestrained specimens.

In order to interpret the tensile test results, the phenomena of failure in the single filament composites must be taken into account. The photomicrographs in Figure 7 show that two different modes of failure in the epoxy may follow tensile failure of the filament. Figure 7A shows a filament failure (at a low value of total strain) which does not propagate into the epoxy; Figure 7B shows a tensile crack in the epoxy emanating from the filament failure. As the amount of inelastic deformation increases, shear failure in the matrix occurs at the broken ends of the filament; and matrix tensile cracks (when they were initially present as in Fig. 7B) grow as shown in Fig. 7D. It is evident that the amount of shear failure is greater where there is no tensile crack (to relieve stresses) (Fig. 7C) than where there is a tensile crack (Fig. 7D). The shear crack at the filament end changes direction (as it grows) so that the new surface being created becomes more nearly normal to the applied load; in absence of tensile cracks, such

*Texaco Experiments, Inc.

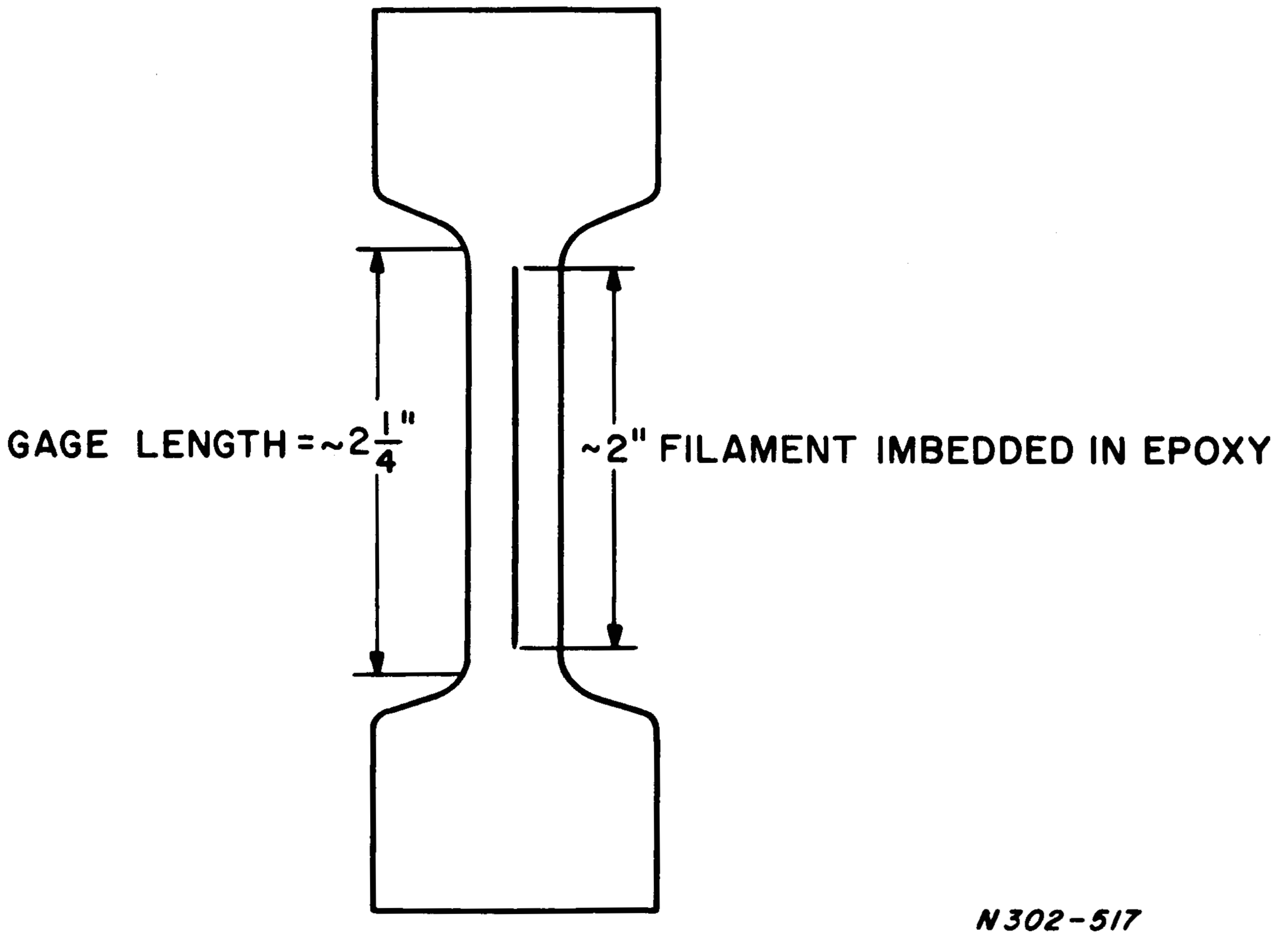


Figure 5. Schematic Representation of a Single Filament Unreinforced Composite.

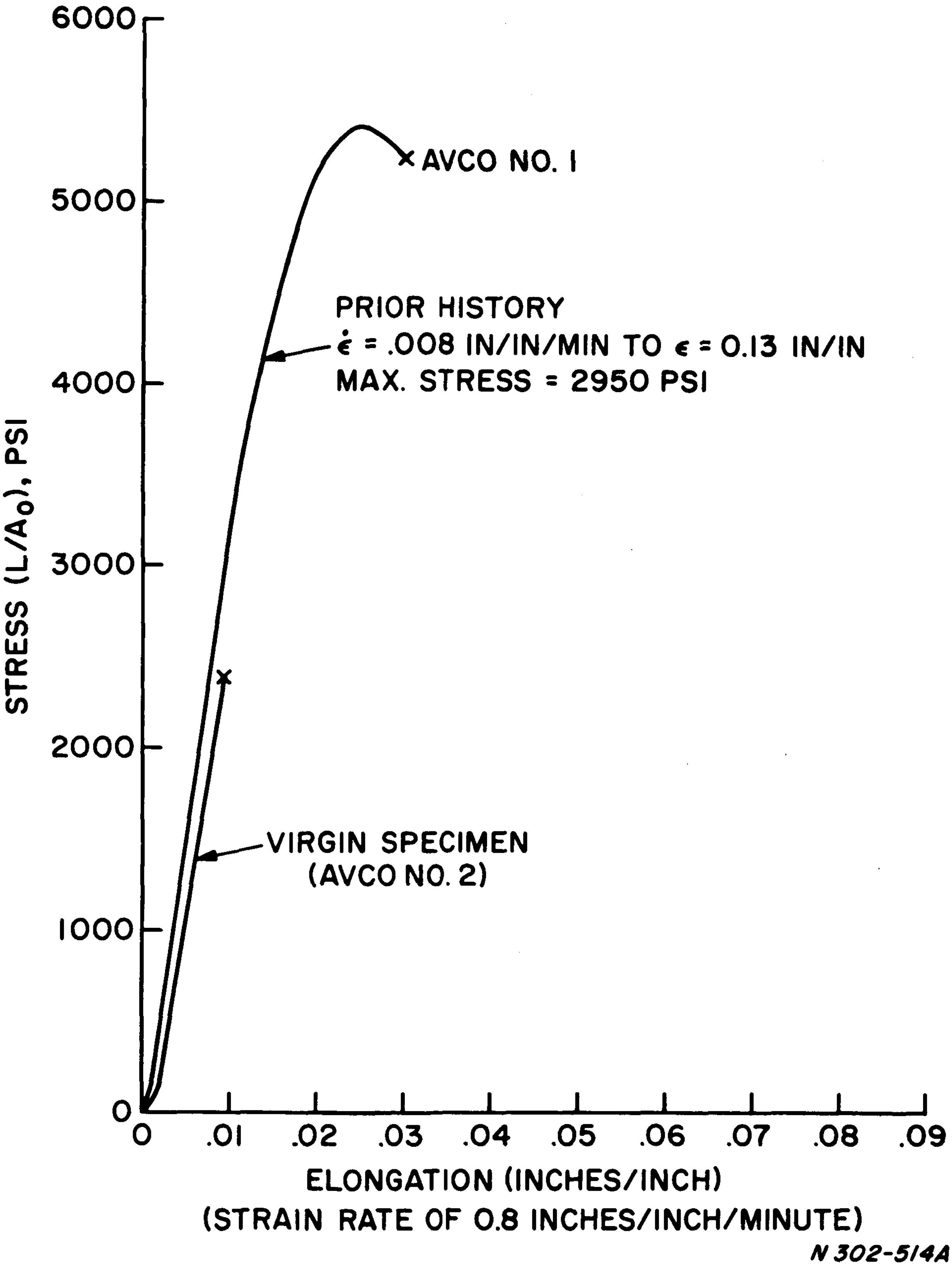
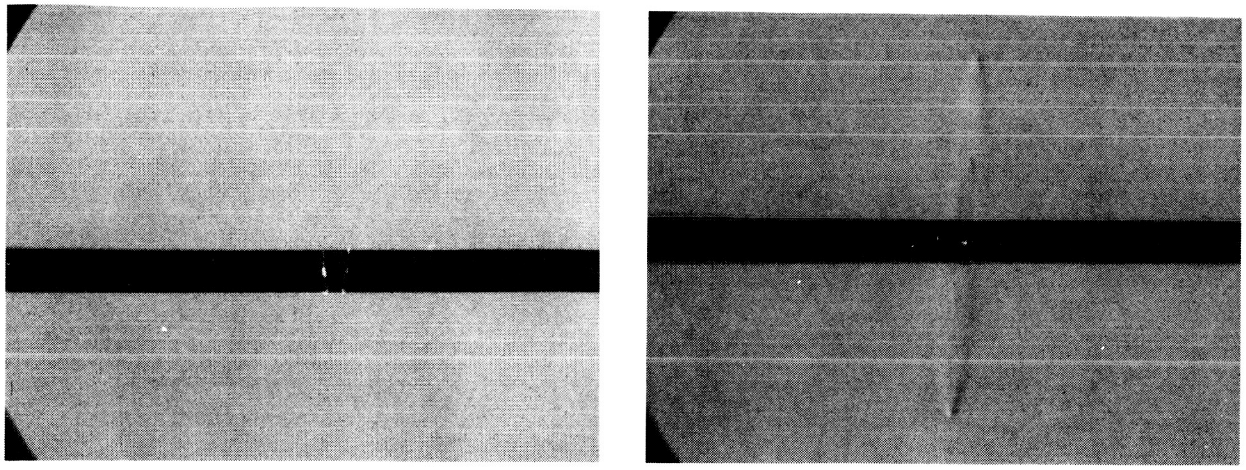
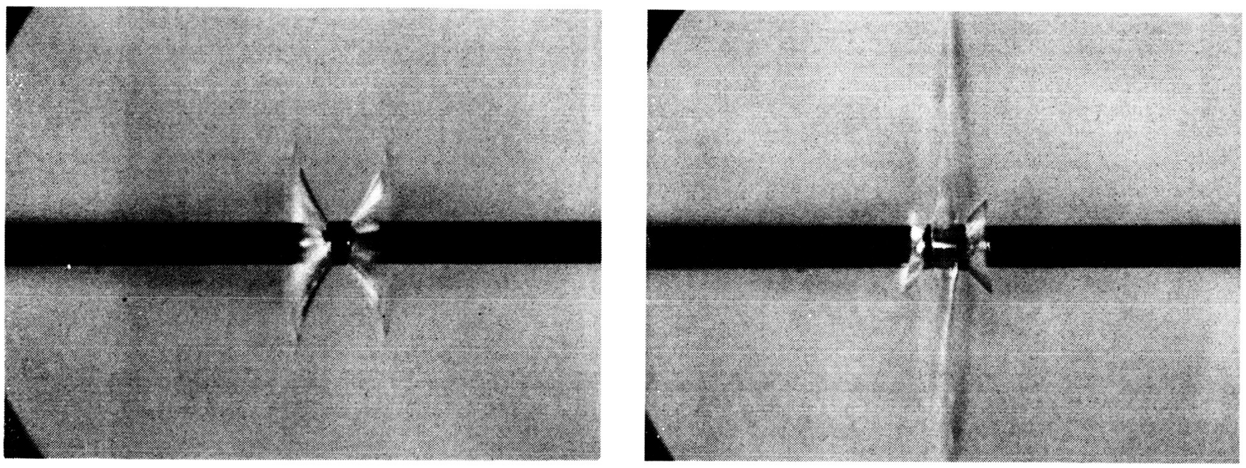


Figure 6. Stress-strain Behavior of Virgin and Pre-strained Epoxy Specimens Strained at 0.8 in/in/minute.



A. NO TENSILE CRACK IN MATRIX　　B. MATRIX TENSILE CRACK

CASE I: HIGH STRAIN RATE, LOW TOTAL STRAIN (AVCO 1)



C. NO TENSILE CRACK IN MATRIX　　D. MATRIX TENSILE CRACK

CASE II: LOW STRAIN RATE, HIGH TOTAL STRAIN (AVCO 2)

Figure 7. Two Consequences of Tensile Cracks in B/W Filaments in Single Filament Epoxy Composites (Oblique Illumination, Longitudinal Views, 58X).

failure would eventually part the entire specimen. This phenomenon (shear failure at fractures in the filament) indicates that the filament-epoxy interface bond is quite good. This shear-type (rather than tensile) failure is, if not the same, at least analagous to the failure mode of "brittle" tungsten in a copper matrix: filament failure is energetically insufficient to cause a tensile crack in the copper and fracture proceeds by a ductile tearing apart of the copper. (When bonding is poor or the epoxy is soft, a different kind of shear failure may occur, namely unbonding along this length of the broken piece.)

Table IV

Tensile Data from Epoxy-B/W/Single Filament Composites

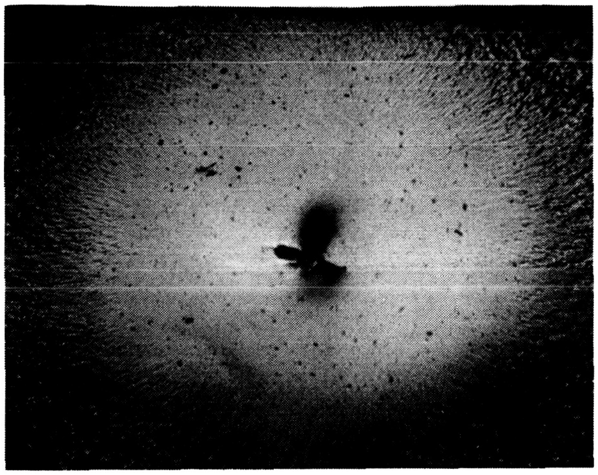
Specimen Identification	Strain Rate (in/in/min)	Stress at Max (Load, psi(1))	Total Strain (at Max Load, in/in(2))	Remarks
AVCO 1	0.8	2380(3)	0.009	7(5) Breaks
TEI 1	0.8	2550(3)	0.010	4(5)
AVCO 2	0.008	3020(4)	0.130(6)	12 Breaks
	0.8	5380(3)	0.03	12(5)
TEI 2	0.008	2800(4)	0.100	13
	0.8	5600(3)	0.04	14(5)
Plain Epoxy	0.8	(7)	(7)	(7)

(1) Load/original area
(2) Cross Head Travel/specimen gage length
(3) Fractured at maximum load
(4) Test stopped, specimen examined for cracks in filament and test resumed at high strain rate
(5) Including break associated with final fracture
(6) Almost all of this chiefly inelastic deformation was recovered soon after unloading
(7) The load exceeded the range being used and the strength was estimated to be between 6000 and 8000 psi with much more elongation than others at $\dot{\epsilon} = 0.8$ in/in/min.

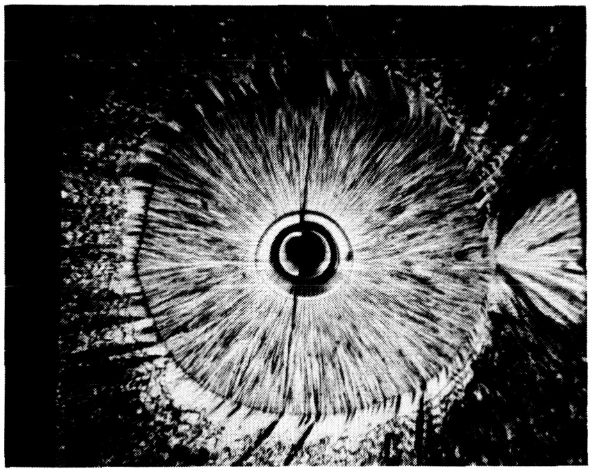
The tensile crack in the matrix in Figure 7B is approximately 0.04-inch in diameter and its area is approximately 2% of the specimen cross-section. This seemed to be the minimum size for non-catastrophic cracks formed in the virgin specimen (at least at the higher strain rate). Thus any crack which forms must initially propagate very rapidly and then slow down and stop. Initially it is very sharp and, by virtue of the intense stress concentration in the matrix at the crack tip, propagation is on essentially a single continuous geometric surface (or plane). As the crack slows down and propagates in a more "ductile" fashion, the stress concentration becomes less, and the slower propagation can then occur on slightly different levels. This should be apparent upon observation normal to the fracture surface. (For example, in Fig. 8D, the smooth inner circle and the radially marked outer circle illustrate the expected consequence when such a change, fast to slow, operates).

As long as the bond strength remains adequate, new and larger cracks will be formed as the load increases. If, eventually, a crack forms which is super-critical in size (large enough, in the initial stage of rapid propagation, to be self-propagating under decreasing load), the specimen will fracture completely. And, because the crack never slows down, the fracture surface will be essentially featureless as shown in Fig. 8A and 8B. (The surface roughness near the free surfaces in Fig. 8A was probably generated by interaction between the advancing crack and the stress pulse reflected from the free surface).

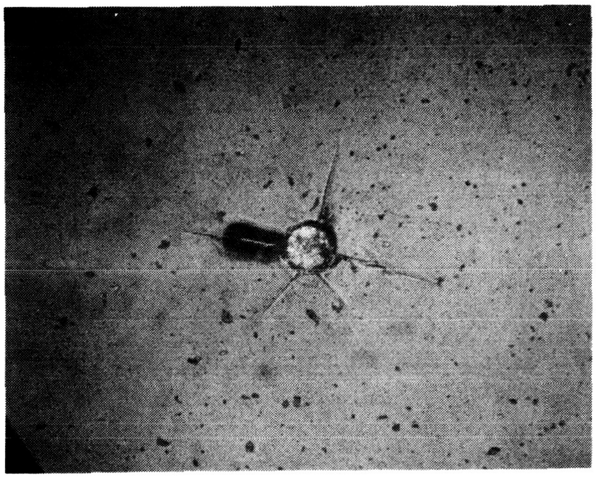
The fracture mode is significantly different at the lower strain rate. The pictures in Figure 8C and 8D show that the initially rapidly propagating crack slows down, over the radially marked region between the concentric circles in Fig. 8D, and stops. In the second test (at the higher strain rate) the crack resumes its advance, still on (the radially delineated) different levels. Because it is thus blunted and because it experiences a gradual increase in load (in contrast to a freshly formed "brittle" crack in the rapid propagation phase), and because the resistance of the epoxy to <u>deformation</u> is markedly rate sensitive, higher stresses are required to move this crack than to keep a freshly formed crack going (as in the virgin specimens, Figs. 8A and 8B). Eventually the crack becomes large enough to so reduce the cross section (a sort of internal necking) that it can continue to grow under



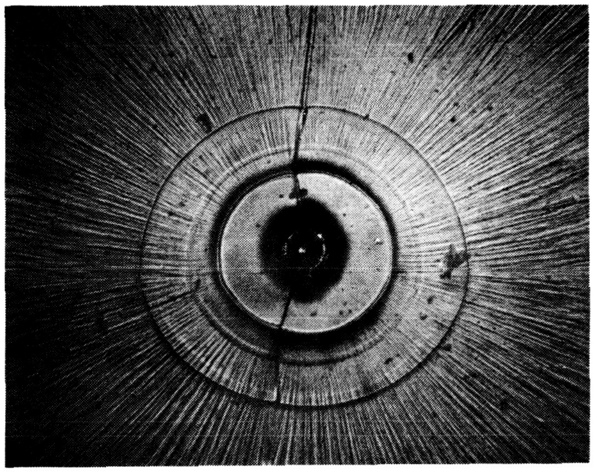
A. AVCO NO. I (17 X)



C. AVCO NO. 2 (17 X)



B. AVCO NO. I (58 X)



D. AVCO NO. 2 (58 X)

CASE I: STRAIN RATE = 0.8 IN/IN/MIN. TENSILE STRENGTH = 2300 psi AT ϵ_L = 0.009 IN/IN. TOTAL OF 4 BREAKS

CASE II: STRAIN RATE 0.008 IN/IN/MIN. CRACKS FORM FROM 1200 psi TO 2920 psi. TOTAL OF 12 BREAKS AT STRAIN RATE OF 0.8 IN/IN/MIN, TENSILE STRENGTH = 5520 psi AT ϵ_L = 0.003 IN/IN

Figure 8. The Effect of Strain Rate History on the Fracture Mode of B/W Filaments in Epoxy (Single Filament Composite).

decreasing load. Finally, when the crack front reaches the outermost position (largest circle in Fig. 8C), its size and sharpness are such as to characterize it as a supercritical crack, which is self propagating.

While the foregoing discussion is not definitely qualified, particularly with regard to its implications for other filaments and matrices, it does suggest an account of behavior consistent with both the tensile test results and the observed fracture mode. It seems incontrovertible that strength, conventional ductility, and the fracture mode are profoundly affected by strain-rate and prior strain history for this very simple filament-epoxy configuration. There is no a-priori reason to suppose that more complicated configurations would be phenomenologically simpler or to suppose that quantitative test results can be predicted on the basis of inadequately sophisticated assumptions. While an epoxy is not a metal with respect to its properties or its detailed mechanical behavior (even epoxies vary greatly), there is again, no a-priori reason to suppose that filament-metal composites will be free of complicated behavior patterns of relevance to performance.

It would be a rather remarkable coincidence if a single continuous linear function, such as the rule of mixtures, turned out to be valid, independent of strain rate, failure mechanism, etc.* In one important respect this is universally recognized in that the rule of mixtures is not expected to apply when the filament-matrix bond strength is inadequate: in this case failure is by "pull out" of the filaments, and the strength is matrix limited. The experiments described here show that, even when bonding is clearly adequate, there is more than one mode of failure having relevance for performance.

The rule of mixtures proposes that, if unbonding does not occur, the properties of a composite will depend upon some statistical distribution

*The failure of mild steel cannot be predicted solely from knowledge of its deformation behavior because low temperatures and/or the introduction of notches introduce a phenomenological transition from ductile to brittle behavior.

of the tensile strength of the reinforcing filaments. It now seems possible to suggest two other relevant modes of failure. The first is failure by the relatively slow propagation of a shear crack originating at a tensile failure in a reinforcing element. Such failures depend upon the filament tensile strength, as the originating component, and upon the properties of the matrix, the medium through which the shear crack is propagated under all of the complicated conditions of constraint imposed by nearby integral filaments. It is suspected that copper-tungsten fails by this mode and it "obeys" the rule of mixtures because of the relatively low scatter in strength of tungsten wire. The second relevant mode is that of the propagation of a brittle crack, originating at a filament failure, through the matrix. Again the properties of the matrix are relevant, but in more than one respect. The resistance to the formation and propagation of a brittle crack is a matrix characteristic and, for closely spaced filaments (in high volume fraction composites) there may be some dynamic attribute of importance. The degree to which a stress pulse, associated with one filament failure, is attenuated as it passes through the matrix to a neighboring filament is a property of the matrix (damping capacity). Composites made using brittle filaments frequently exhibit only one break per filament (see later discussion) and this may indicate that the matrix readily propagates a brittle crack or that it does not attenuate the shock pulse associated with first failure and thereby propagates a concentrated stress pulse.

The foregoing is necessarily incomplete and speculative. Its purpose is to suggest that failure to enjoy the expectation of the rule of mixtures may not always be associated with poor bonding or fabricating difficulties. It does suggest that several properties of the matrix, both static and dynamic, are of possible relevance for discontinuous (and continuous) reinforcement.

(c) Tensile Test Results of Continuous-Filament/Epoxy Composites

Thus far a series of three epoxy-SiC/W continuous composites has been fabricated and tested. The results are shown in Table V. No attempt was made to hypothesize on the strength behavior of these small samples.

However, the mode of fracture was examined, and it became obvious that all of the filaments broke at a common plane in the specimen. This is an unexpected result because of the rather dilute nature of the composites and also because of the possibility of a statistical failure mechanism which is usually inherent in brittle materials.*

It has been tentatively concluded then that the first fiber break that occurs in the composite is responsible for its failure. Of course, the SiC/W fibers used here are remarkably similar in strength on an individual basis when compared compared to B_4C/B/W or B/W filaments.[11] Such behavior then might be explained except that the evidence that no other breaks are ever observed is a rather stringent requirement implying that all the filaments used in the composites made thus far are essentially equal in strength. It is more likely then that a shock impulse from a failed fiber (as discussed earlier) can be transmitted through the matrix to its neighbors and then in turn fail thereby leading to an avalanche effect to composite failure.

Table V

Tensile Results of Epoxy - SiC Continuous Filament Composites

Spec. No.	No. Filaments	Volume Percent Filaments	Tensile Strength (psi)	Calculated Tensile Strength (psi)[a]
48	36	3.1	17,300	12,920
50	14	1.6	7,330	8,150
51	17	5.3	18,100	20,060
56	0	0	2,920	2,920

[a] Calculated using the rule of mixtures and ignoring the matrix contribution

Figure 9 is a photograph at 16X which illustrates this behavior for a three-filament composite. The center filament appears to have failed first and "shocked" the two adjacent filaments which in turn also failed.

*A mathematical and experimental treatment of statistical failure behavior is well documented for epoxy-fiber glass composites by Rosen.[13]

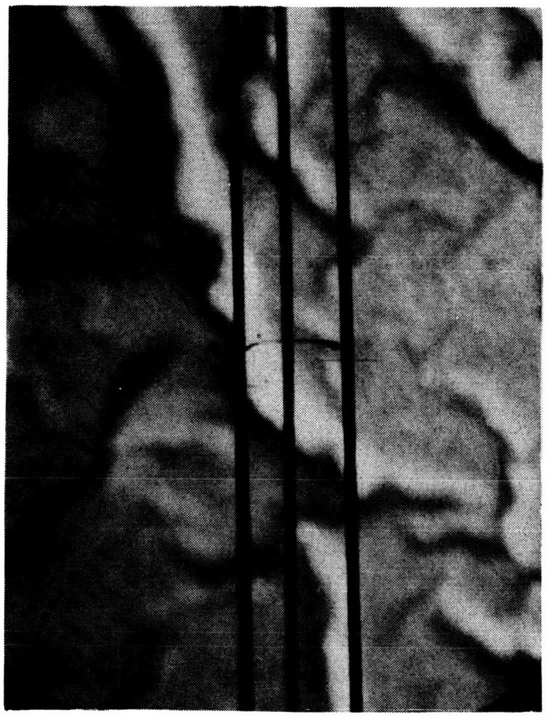


Figure 9. Three Filament Epoxy-SiC/W Composite Illustrating Multiplication Effect.

2. Aluminum - Fiber Composites

(a) Aluminum - Single Filament Composites

Because the possibility existed that the first quarter work[(2)] using the plate, tube and filament techniques for fabricating composites could be in error, a duplicate study was made using an aluminum powder technique already described in another section of this report. A $B_4C/B/W$ filament was imbedded into hot pressed aluminum, strained to 6 percent at room temperature, digested in 50-50 $HC\ell$ -H_2O and the resulting pieces counted. A summary of the data is presented in Table VI. No apparent difference in the data is noted so that the techniques appear to be directly comparable since a different fabrication technique produced essentially identical results. It is felt that greater confidence can be placed on data derived from this experimental technique.

A study of hot pressed aluminum-SiC/W single filament specimens was also made to duplicate the experiments performed on $B_4C/B/W$ filaments during the last quarter. A summary of these results is also presented in Table VI. The data show that, as in the case of $B_4C/B/W$ filaments, the SiC/W filament break-up is essentially complete at quite small strains. The three tests were made using an extensometer attached to the specimen gauge length (tests 11-13), and from these the quantitative relationship between strain and the extent of break-up was established, as with the $B_4C/B/W$ case. Strains of less than 1% (total) did not cause filament breakage, while strains of 2% total resulted in the maximum breakage ($\sim$ 32 pieces). Based on an average strain of 1-1/2% and an elastic modulus of SiC approximately of 60×10^6 psi, this experiment indicates the SiC filaments $\sim$0.1" long also had an average tensile strength of 900,000 psi and that, therefore, the shear strength of the strain hardened aluminum adjacent to the filament was $\sim$18,000 psi based on the relationship $L_c/d = \frac{\sigma}{2\tau}$. The results parallel those of the $B_4C/B/W$ filaments even though the SiC filament tensile values determined by individual testing were slightly lower and less scattered. It is to be noted however that tensile fracture in such composites is always

Table VI

Filament Break-up in Strained Unreinforced $B_4C/B/W$-Hot Pressed Aluminum and SiC/W-Hot Pressed Aluminum Composites

Test No.	Filament Type	No. and Length of Filaments*	Plastic Strain of the Composite (%)	Remarks on Filament Fracture
10	$B_4C/B/W$	1 2 inch length	6	40 pieces
11	SiC/W	1 2 inch length	1	0 pieces
12	SiC/W	1 2 inch length	1 1/2	9 pieces
13	SiC/W	1 2 inch length	2	32 pieces

* Prior to testing of Composite

obtained on extremely well aligned "specimens" having very short gauge lengths. Such test conditions can only be approximated during individual testing of filament materials.

(b) Aluminum-SiC Continuous Filament Composites

Studies of continuous filament composites of Al-$B_4C/B/W$[2] had shown that efficient reinforcement was possible, at least with the approximately 40 volume percent filaments then used. A more extensive program was initiated using SiC/W filaments in an attempt to more fully study the continuous reinforcement of aluminum as a function of volume fraction. The decision to use SiC/W filaments was made to simplify the handling of large quantities of filament, because the $B_4C/B/W$ filaments presently on hand are less than 0.0027" in diameter, while the SiC/W filaments average 0.004" in diameter. Accordingly, a large number of 2" long, 0.004"-diameter SiC filaments were cut for use in a liquid aluminum infiltration apparatus. Infiltration methods were the same as previously used[2], including both the infiltration assembly and the actual infiltration unit. A new specimen mold was designed which is compatible with the infiltration assembly and allows the simultaneous infiltration of five specimens. This mold is shown in Figure 10. The mold cavities are 0.062" in diameter and 3" deep so that final specimen diameters using 2" long filaments would be 0.062" diameter by 2" long. Before proceeding further however, as a check on the wettability

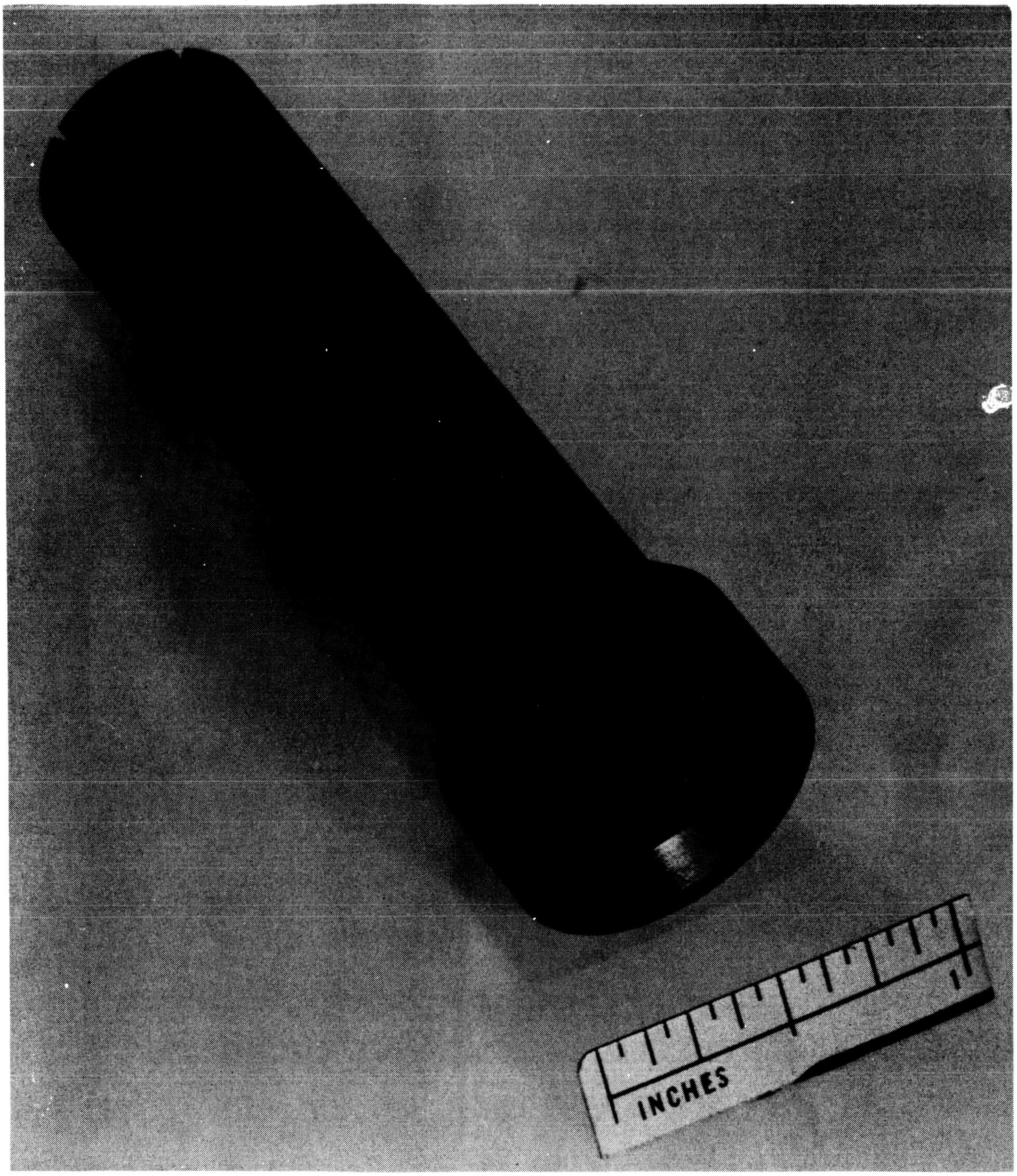


Figure 10. Multiple - Specimen Mold Used to Infiltrate Five Composites Simultaneously.

of SiC/W filament by molten aluminum, a preliminary infiltration was made in a single composite containing 32 v/o filaments in the "as received" condition. A casual examination of the outward appearance of the resulting composite showed a sound specimen and good "wetting", but a metallographic examination of the fracture surface of the specimen broken in tension showed void areas along the filament (see Figure 11). Thus, it appeared necessary to promote wetting with metallic coatings similar to those which were found necessary during former work.[2] Accordingly, about 500 2"-long SiC/W filaments were given a thin sputtered coating of titanium followed by a sputtered coating of nickel. Filament loadings of 5, 10, 20, 40 and 60 volume percent were then prepared by packing the filaments into the five cavity graphite mold and infiltrated with molten aluminum at 720°C. The composites were held at this temperature for 10 minutes to insure complete infiltration and then allowed to cool rapidly by passing cool hydrogen gas over the infiltration assembly. This technique produced excellent, fully dense aluminum-SiC/W filament composites.

Two such runs were made. The composite specimens were then carefully removed from their molds and identified for subsequent tensile testing at room temperature. Special gripping of the specimens was necessary to minimize composite damage and mis-alignment. A schematic diagram of the tensile specimen design is shown in Figure 12. All specimens had a 1/2" gripping length and a one-inch gauge length.

Tensile test results are presented in Table VII. Tensile tests were performed on an Instron tensile testing machine at a strain rate of 0.02"/" min. An analysis of the data indicates that possible damage to the filaments had occurred as a consequence of the thermal processing necessary to form composites. In order to elucidate these results the composites and their individual components were examined to include: (1) room temperature tensile testing of individual filaments as-coated and after thermal cycling to duplicate the thermal history of the fibers during composite fabrication, (2) examination of the interfacial reaction of titanium/Ni coatings on SiC/W

Figure 11. Voids in SiC - Aluminum Continuous Composite - No Coating

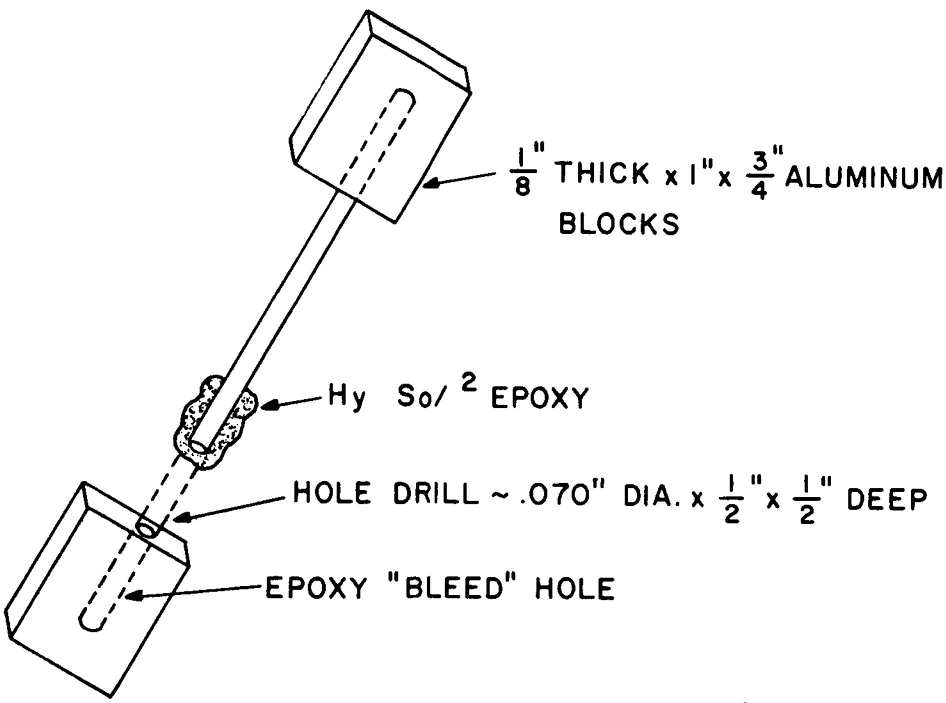


Figure 12. Schematic Diagram of SiC/W - Aluminum Tensile Specimen.

Table VII

Room Temperature Tensile Strength of SiC/W-Aluminum Containing Composites

Spec. No.	Load (Lbs)	σ (psi)	v/o Fibers	σ Theoretical* psi	Eff. %
1-uncoated	220	60,000	32	105,000	60
2-Pi/Ni	76	21,200	5	16,500	130 (1)
4-Ti/Ni	31	8,670	10	33,300	26
6-Ti/Ni	59	16,600	20	66,600	25
8-Ti/Ni	79	22,000	40	133,200	16.5
10-Ti/Ni	193	53,300	60	199,800	26.6
3-Ti/Ni	20	5,600	5	16,500	33.8 (2)
5-Ti/Ni	25	7,000	10	33,300	21
7-Ti/Ni	105	29,400	20	66,600	44
9-Ti/Ni	120	33,500	40	133,200	25
11-Ti/Ni	193	53,300	60	199,800	26.6

*Based on as-coated strength of filaments using rule of mixtures, ignoring matrix contribution

(1) Anomaly due to matrix contribution

(2) Specimen damaged on loading. Tensile strength of matrix alone.

filaments using electron probe techniques, and (3) examination of the fractured area of the composites by removing the aluminum matrix by etching in 50-50 HCl -H_2O.

Figure 13 shows the results of thermal cycling on the strength of coated SiC/W filaments. It is to be noted that there is a serious loss in strength of the same order (~75%) as was indicated by the room temperature tensile strength of SiC/W-Aluminum composites reported earlier. Uncoated specimens showed essentially no change in strength as a result of thermal cycling.

For electron probe examination of SiC/W filaments, filaments were coated with titanium and then heat-treated under the following conditions: 1150°C, 3 hours, 8×10^{-5} torr. Examination of a fractured end of such a filament, (Figure 14) revealed that: (a) silicon had moved from the silicon carbide region in two directions - toward the tungsten substrate as well as into the periphery of the filament, i.e. the coated region. Evidence was also noted (Figure 14) that titanium had diffused into the silicon carbide deposit but only to a minor extent. It is not clear whether the small spot of highly concentrated titanium (shown at about 4 o'clock in Figure 14-D) is the result of diffusion, or whether the titanium coating flaked off during testing of the filament. Thus it is clear that weakening of the filaments is due to mobility of silicon in the presence of titanium since without the coating the same conditions produced little weakening.

An examination of the fractured area of the SiC/W-aluminum composites was made by etching away the aluminum (See Figure 15) and noting the debris which remained. Observations were that first all filaments in all the composites broke at the same area within the composite. Secondly, only a minute amount of debris remained after etching. These results were somewhat unexpected since other investigators[13] had shown that statistically, many failures within each filament would be expected before final failure of the composite is initiated. The results here do not show this accumulative type failure mechanism but imply that one filament failure could be responsible

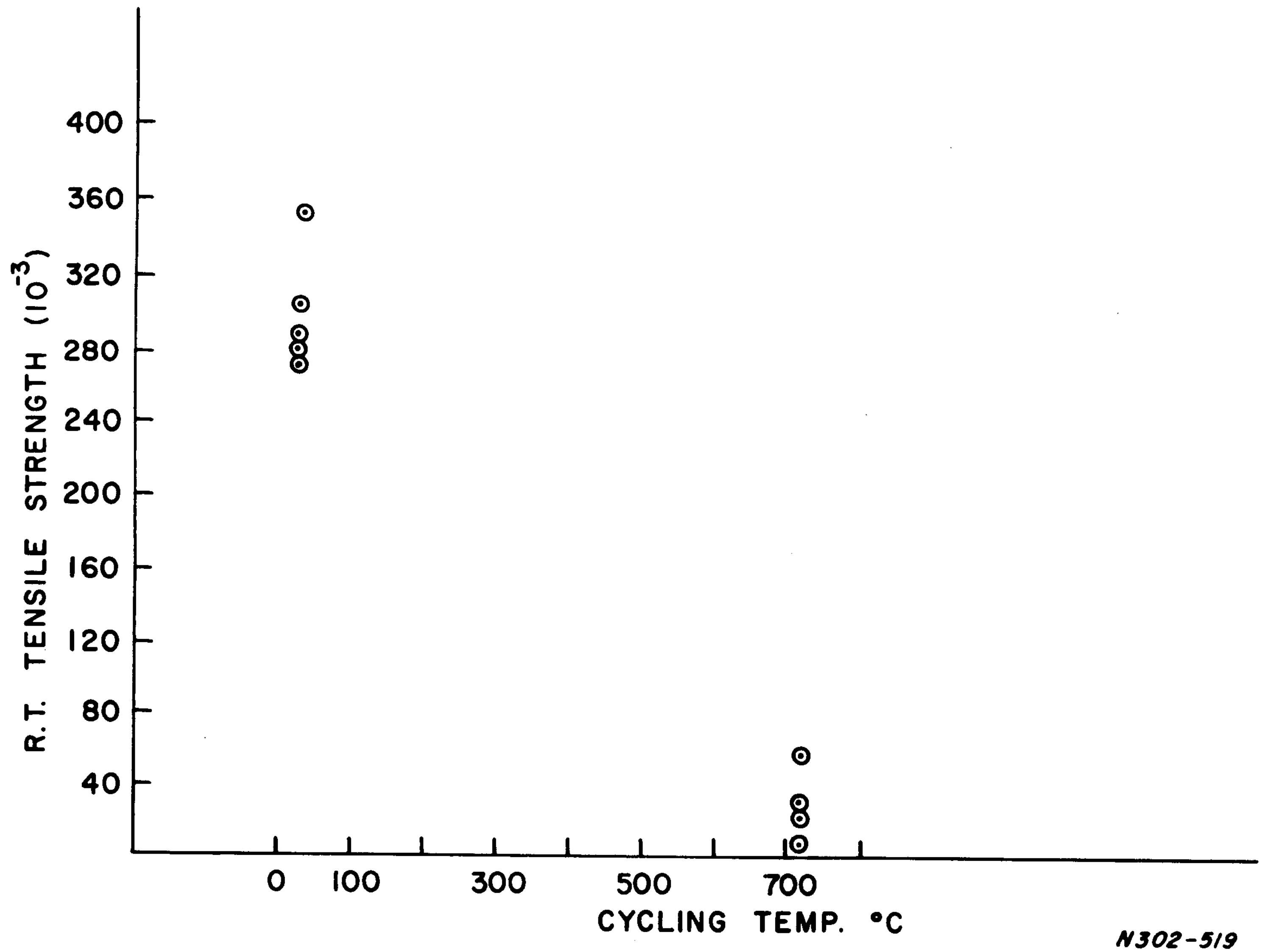
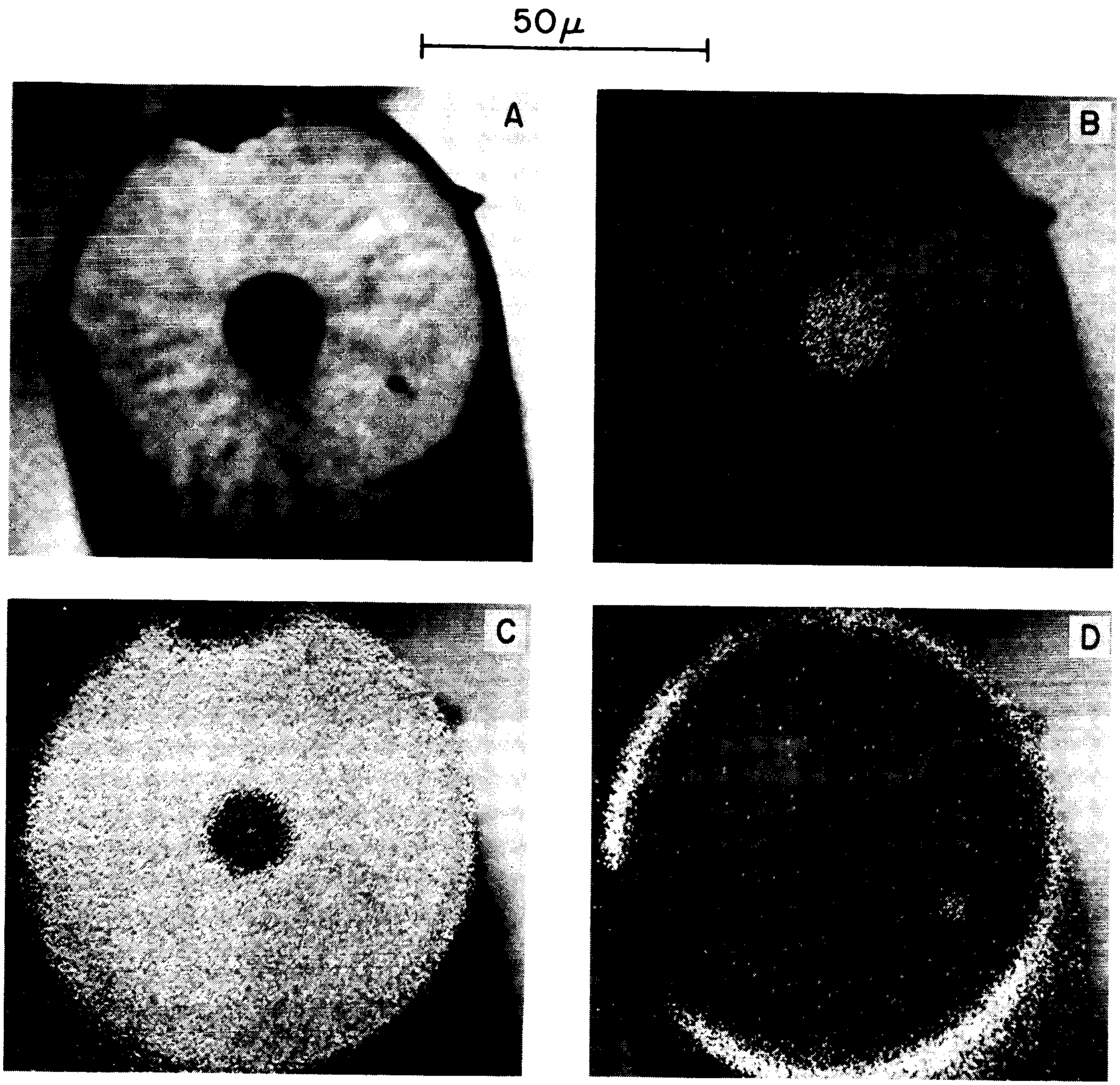


Figure 13. Effect of Thermal Cycling on the Room Temperature Tensile Strength of Ti/Ni Coated SiC Filaments.



A. - SPECIMEN CURRENT IMAGE (I_s)

B. - TUNGSTEN X-RAY IMAGE + I_s

C. - SILICON X-RAY IMAGE + I_s

D. - TITANIUM X-RAY IMAGE + I_s

Figure 14. SiC/W Filament with Ti Coating after Heat Treatment (870X) (Current and Composite X-ray Images).

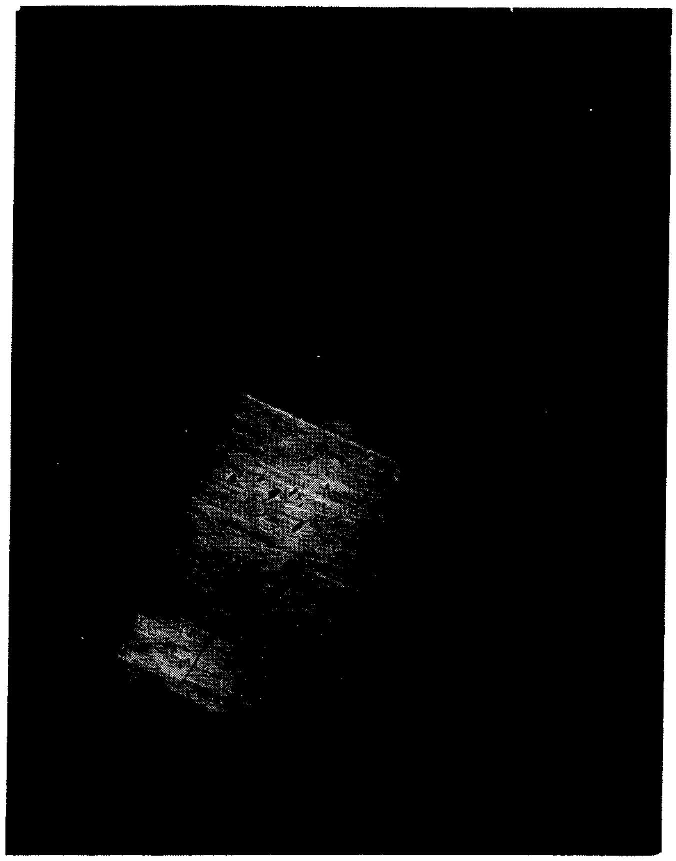


Figure 15. Fractured Area of SiC - Aluminum Composite after Etching.

for the composite failing as an autocatalytic effect. This observation that the matrix can transmit failure impulses to adjacent filaments has also been noted for epoxies as discussed earlier in this report.

III. CONCLUSIONS

1. Growth of boron carbide whiskers by reduction of boron tribromide and carbon tetrachloride appears to be inhibited by insufficient nucleation; the reduction and deposition of boron carbide occur quite readily. Futher experiments are in progress in an effort to prenucleate deposition surfaces.

2. Deposition of boron carbide layers of controllable thicknesses on tungsten core-boron filament substrates have been achieved with little difficulty, except in the controlling of electrical power to the filament. This is being remedied and production of sufficient $B_4C/B/W$ filaments for further micromechanical studies is expected to begin in the near future. Optimization of properties will be conducted in connection with production runs.

3. Material characterization studies on both matrix materials and filaments have shown that (a), aluminum powder processing can produce single filament tensile specimens which give the same results as those achieved previously[(2)] using a hot pressed sheet and tube method; (b), $B_4C/B/W$ filaments tested at 0.1" gauge length showed a maximum strength value of 540,000 psi, near the calculated strength of the filaments when the relationship $\frac{L}{d} = \frac{\sigma}{2\tau}$ was used in conjunction with the aluminum-single filament work; (c), tensile strength vs. gauge length results for SiC/W filaments show less scatter in individual tests and less dependence on gauge length than either $B_4C/B/W$ or B/W filaments. It is concluded that the fracture strength of this material is controlled by a notch type defect similar to that which operates for cast iron; (d), SiC/W filaments when coated with titanium and heated are weakened-electron probe analysis of cross section of one of these coated SiC/W filaments showed that an interaction of silicon with tungsten occurs which is promoted by titanium, while no reaction occurs if titanium is eliminated; (e), an epoxy-Novalac formulation has been developed which has significant "ductility" and bonds well to $B_4C/B/W$, SiC/W and B/W filaments.

4. Continued work on single filament-aluminum composites has shown that similar results are obtained with $B_4C/B/W$ and SiC/W filaments when the fabrication of the composites is changed to a powder processing technique. The data still indicate that filament fracture occurs at about

1-1/2% strain for both types of filaments. Calculated strengths using this magnitude of strain suggests that strength values of the order of 600,000 to 900,000 psi are possible for filaments about 0.1 inch long. These strength figures seem reasonable if ideal alignment and gripping of the individual filament pieces within the composite are considered.

5. Continuous-filament composites of aluminum-SiC/W and Epoxy-SiC/W filaments have been fabricated. It was found that sputtered coatings on the SiC/W filament are necessary to promote wetting and to insure sound aluminum based composites. Unfortunately, the coatings chosen, Ti/Ni, (which were excellent for the $B_4C/B/W$ filaments) drastically reduced the strength of the filaments during composite fabrication.

6. Studies of the fracture of both metal and epoxy continuous-filament composites have shown that a statistical fracture mode does not seem to apply, since it appears that all filaments in the composites broke in one plane. This suggests that one filament fracture is sufficient to cause fracture of its neighbors in an autocatalytic manner.

7. It has shown that the fracture mode, the strength, and the "ductility" of single-filament epoxy composites are markedly affected by the strain rate and prior strain history of the specimens.. An argument is advanced that the rule of mixtures does not deal with these variables of dynamic origin (other than unbonding) and is therefore inadequate to apply to these other modes of composite performance.

IV. FUTURE WORK

Future studies will continue to focus attention on producing $B_4C/B/W$ filament for composite studies, and additional experiments concerning production of boron carbide whiskers by chemical vapor deposition rather than by evaporation. Further studies of the fracture behavior of both single-filament and continuous-filament composites in both epoxy and aluminum will be pursued to further clarify the present hypothesis of both filament and composite failure. Experimental work on achieving discontinuous reinforcement will also be started. Candidate materials will be studied to find a suitable metal coating which will promote the wetting of SiC/W filaments with molten aluminum so that interaction or weakening of the filaments will not occur.

REFERENCES

(1) A. Gatti, J.B. Higgins, R. Cree, E. Feingold, "Boron Carbide Continuous Filaments", AFML-TR-65-354, Part I, Nov. 1965.

(2) A. Gatti, J.M. Berry, J.J. Gebhardt, E. Feingold, "Investigation of the Reinforcement of Ductile Metals with Strong, High Modulus Discontinuous, Brittle Filaments, First Quarterly Report, NASw-1543, Feb. 1967.

(3) A. Gatti, R. Cree, E. Feingold, "Synthesis of Boron Carbide Filaments," NASw-670 Final Report, July 10, 1964.

(4) A. Gatti, C. Mancuso, E. Feingold, R. Mehan, R. Cree, "Study of the Growth Parameters Involved in Synthesizing Boron Carbide Filaments," NASA CR-251, July 1965.

(5) A. Gatti, C. Mancuso, E. Feingold, R. Jakas, "Study of the Growth Parameters Involved in Synthesizing Boron Carbide Whiskers, " NASw-1205, Final Report, March 1, 1966.

(6) A. Gatti, E. Feingold, J.J. Gebhardt, J. Berry, V.A. Cordua, "Study of the Growth Parameters Involved in Synthesizing Boron Carbide Whiskers," NASw-1383, Final Report, November 1966.

(7) D.L. McDanels, R.W. Jech and J.W. Welton, "Metals Reinforced with Fibers," Metal Prog., Vol. 78, No. 6, Dec. 1960, pp. 118-121.

(8) J.B. Higgins and J.J. Gebhardt - Presented at the Electrochemical Society Meeting, Dallas, Texas, May 8, 1967.

(9) J.B. Higgins, E. Feingold, A. Gatti, "Boron Carbide Continuous Filaments", AFML-TR-65-354, Part II, Aug. 1966, pp. 35.

(10) J.J. Gebhardt, unpublished data.

(11) J.M. Berry, Task 6, "Research on Improved High-Modulus, High Strength Filaments and Composites thereof." Sept. 1965, General Electric Company, U.S.A.F. Contract AF 33(615)-2126 Technical Report AFML TR-65-319.

(12) V. Mazzio, unpublished research.

(13) B.W. Rosen, "Tensile Failure of Fibrous Composites." AIAA 2 No. 11, Nov. 1964.

ACKNOWLEDGEMENTS

Acknowledgement is given to Messers. V. Mazzio and V. Diano for their valuable assistance concerning epoxy technology and especially to Mr. R. Mehan for his suggestion on epoxy-single filament composites as a means of studying composite failure mechanism. Also thanks to Dr. W.H. Sutton who reviewed the manuscript.